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A Novel Mechanical Method to Measure Shear Strength in Specimens Under Pressure

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ABSTRACT

A new experimental apparatus has been developed for performing shear tests on specimens held under moderately high hydrostatic pressures (on the order of 4 GPa). This testing procedure experimentally determines the pressure-dependent shear strength of thin foil specimens. The experiments provide calibration data for models of materials subjected to extreme pressures such as the Steinberg-Guinan hardening model and can assist in model validation for discrete dislocation dynamics simulations, among others. This paper reports the development of the experimental procedures and the results of initial experiments on thin foils of polycrystalline Ta performed under hydrostatic pressures ranging from 1 to 4 GPa. Both yielding and hardening behavior of Ta are observed to be sensitive to the imposed pressure.

INTRODUCTION

Plastic deformation in metallic systems occurs primarily via dislocation generation and movement due to shear stresses. In the case of hydrostatic loading no shear stresses are present to provide a driving force for dislocation motion, therefore structural properties are not changed due to hydrostatic pressure alone. Nevertheless it is found in the literature that properties such as hardening and ductility of metals are sensitive to superimposed pressures. Even at relatively low pressures of 0.7-3.0 GPa a remarkable increase in ductility of some materials has been reported [1]. Bridgman's results [2,3,4,5] suggest that pressure hardening occurs in Mo, Ta and Ni.

Another property of interest is the shock-induced phase transformation. Numerous cases of phase changes have been reported when materials were subjected to pressures exceeding 30 GPa [6,7,8]. Finally from the physics perspective, the relationship between material strength, elastic constants, and microstructure is of great interest, and can lead to new insights of mechanisms of plastic flow under conditions of imposed pressure.

Most high-pressure research has been carried out under static conditions. For pressures in the range of 0-3 GPa testing has been conducted using a variety of media including solid, liquids and gases [1]. To achieve higher pressures, experiments have been conducted using the diamond anvil cell, [6], where the specimen is loaded to high pressures between the diamond anvils. Although this device allows ultrahigh pressures to be reached readily, it has the deficiency that the hydrostatic, frictional and deviatoric stresses increase in an uncontrolled manner as the load increases, making it difficult or impossible to determine strength and work hardening from experimental results. Another disadvantage of this method is that typically the volume of material tested in these kinds of systems is small and the properties that are observed may be functions of sample size. Postmortem analysis in these types of experiments is often difficult because of the small specimens necessarily used.

THEORY (OR EXPERIMENT)

In order to overcome these inconveniences a new test procedure, similar to that established by Bridgman [2-5], has been developed to investigate materials response in a pure shear deformation under high pressure conditions. This system is depicted in Fig 1. This test emphasizes two major features: strict control in the loading path, in order to separate the effects of hydrostatic and deviatoric stresses, and the ability to perform high pressure experiments using a larger specimen size that can be analyzed using standard characterization tools subsequent to testing, such as hardness measurements and TEM analysis.

In this modified Bridgman cell, three independent supported tungsten carbide anvils were used (figure 1 b). The anvils in the top platen are formed with a hemispherical section that fits snugly into the mating surface. Using a thin foil of indium between these hemispherical surfaces provides for self-leveling of the anvils upon initial loading. The same pressure is attained between all anvils by positioning each anvil atop a hydraulically-controlled piston, all of which are connected to a common oil reservoir in order to establish equivalent load on all anvils. Extensometers were attached to the anvils to measure the local strain on the sample being deformed and to avoid issues of machine compliance.

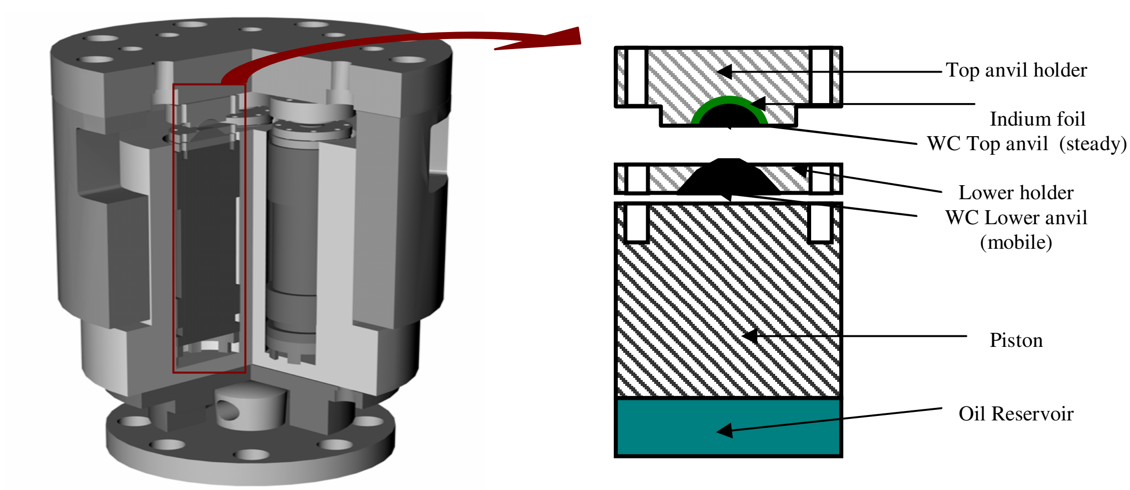

Figure 1. Schematic of the modified Bridgman cell used to perform the experiments.

The deformation of the specimens in the tri-anvil apparatus is achieved with the same general philosophy as with the original Bridgman design as shown in figure 1: axial load followed by rotational displacement.

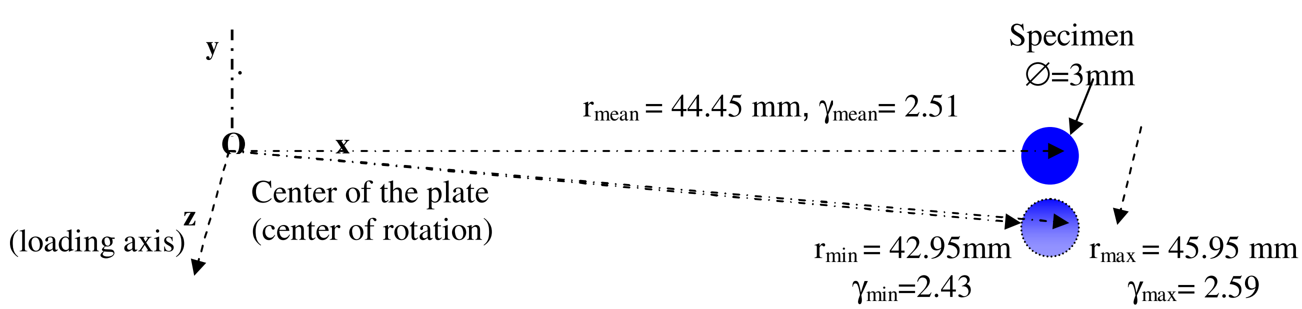

Figure 2. Top view of the arrangements of the anvils in the lower

In this new experiment, the anvil centers are positioned on a circle centered on the loading axis of the testing system (figure 2). This arrangement produces a near negligible shear strain gradient, in fact this shear loading can be assumed unidirectional because of the small specimen size, (radius/thickness ratio ranges from 60 – 600) and low angle rotations ($\theta \approx 0.5^{\circ}$ = 8.7 mrad) in comparison to the circumference of the circle on which the specimens lie R ≈ 44.45 mm, a rough calculation leads to a value for the strain gradient of 6 % from end to end along the specimen thereby creating a more uniform shear stress in the specimen as compared with the original Bridgman single anvil design where the strain is radial function from the center of the specimen.

DISCUSSION

For this experiment it is crucial to determine whether the desired state of hydrostatic pressure was be attained by application of axial load on the thin foil specimens between the two anvils. The aim is to have the major portion of the specimen under a uniform hydrostatic pressure. By using thinner foils, a more uniform distributed pressure can be achieved, but if the thickness is too small, the possibility exists that contact could occur between the anvils during specimen testing thereby confusing the measurement of load on the specimen. Assuming proper platen alignment, this contact could occur by either of two processes: elastic deformation of the anvils due to bearing stress, or specimen conformity to the roughened anvil surfaces.

To experimentally find the optimum specimen thickness, a set of Ta foils purchased from a commercial vendor and vacuum annealed at 1000C with thicknesses of 50, 25, 12.5 and 5 μm were used for the initial experiments described herein, the samples were disks of 3 mm in diameter. By using specimens of this size a complete post-mortem characterization was possible in order to validate the adequacy of the testing device. The stress-strain response is shown next in figure 3.

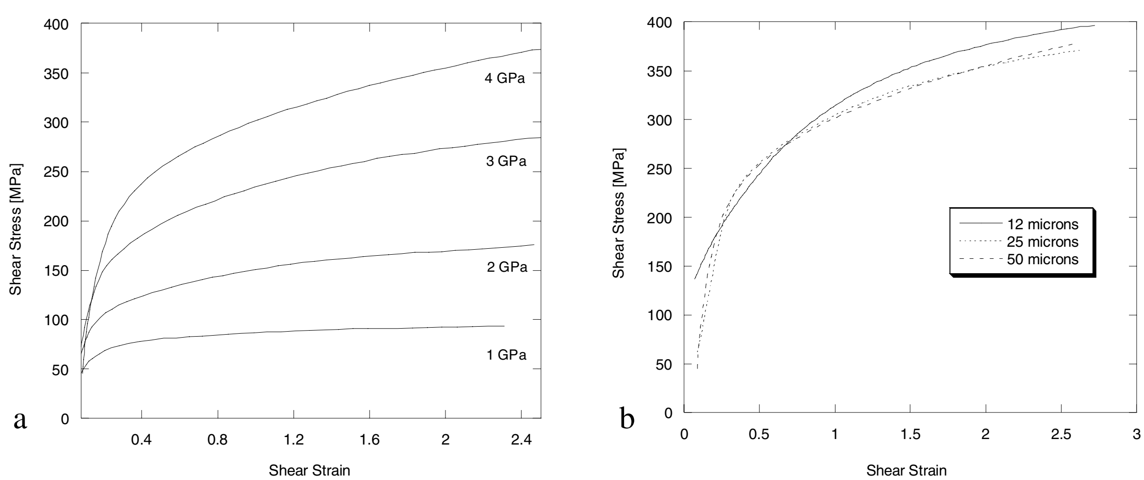

Figure 3. Stress – strain results for: a) sample with thickness of 50 microns, b) response of specimens with different thicknesses.

The samples with thicknesses of 25 and 12.5 microns show a similar response as the 50 micron samples, as it is depicted in figure 3b for a pressure of 4 GPa, the stress-strain response converged being independent on the thickness of the sample. The results of the 5 microns thick sample have been determined to be erroneous, it was established that these samples caused platen contact and binding due to elastic strain in anvils and test samples added to the inherent roughness of the anvils.

Hardening

A second issue imperative to the establishment of this new experimental technique is the determination that the stress state in the material upon initial loading is primarily hydrostatic. As it has been mentioned plastic deformation occurs primarily via dislocation generation and movement due to shear stresses. In the case of hydrostatic loading no shear stresses are present to provide a driving force for dislocation motion, therefore structural properties are not changed due to hydrostatic pressure alone. In addition, dislocation activity causes strain hardening in the material, so if significant dislocation activity occurs, the hardness of the metal after loading is expected to increase. To investigate this phenomenon hardness measurements were made across the specimens on the different thicknesses subjected to pressures of 4 GPa, the maximum attained during the tests, the results of the 12.5 microns samples are shown in figure 4.

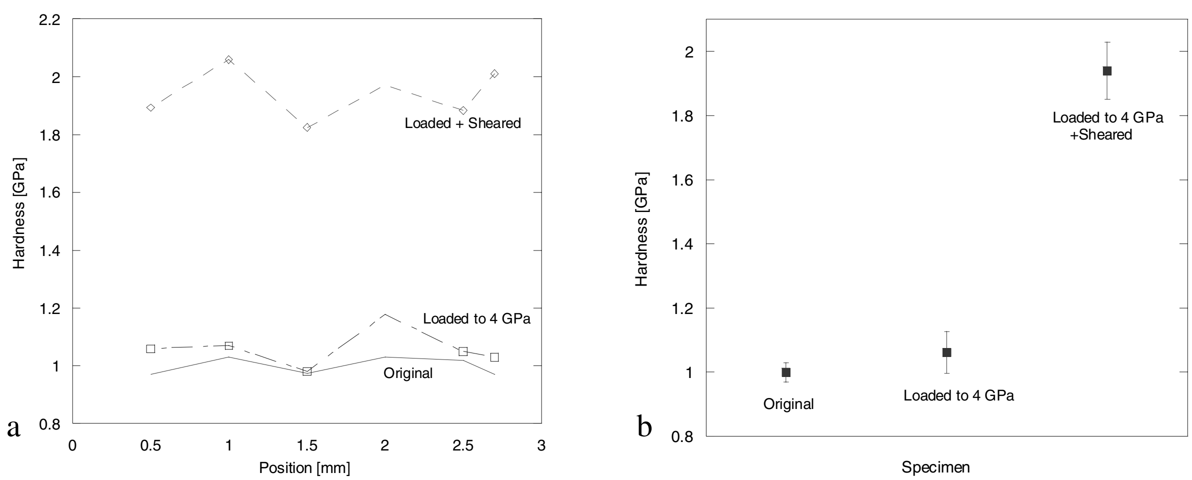

Figure 4. Hardness of specimens under 4.2GPa.

Hardness measurements were made every 0.5 mm from side-to-side across the specimen diameter. It is evident from figure 4 that the samples subjected to loading show no hardening effects due to the superimposed pressure, the original value was estimated to be 1.02±0.03 GPa, and after loading equal to 1.08± 0.04GPa, the two values being the same within the experimental error when measuring hardness. On the contrary, the samples sheared after loading show values of 1.97± 0.09 GPa, an evident increase in hardness close to 82%. These values of hardness corroborate that the stress-strain response is a measure of the strength of the specimens held under pressure and not an effect of *a priori* deformation caused by axial loading.

<u>TEM analysis</u>

Bright field TEM was done in order to reveal microstructural changes. This analysis was done on both the deformed and undeformed specimens. Figure 5a shows the image of the original Ta foil that was used for the tests. No appreciable dislocation density prior to the deformation process is observed. For the specimen loaded/unloaded to a pressure of 4.2 GPa without shearing (figure 5b), a slight increase in dislocation content is observed, but the grains are still relatively free from dislocation debris. This explains why the measurements of the hardness in the specimens loaded and unloaded without shearing were close to those obtained for the undeformed specimens.

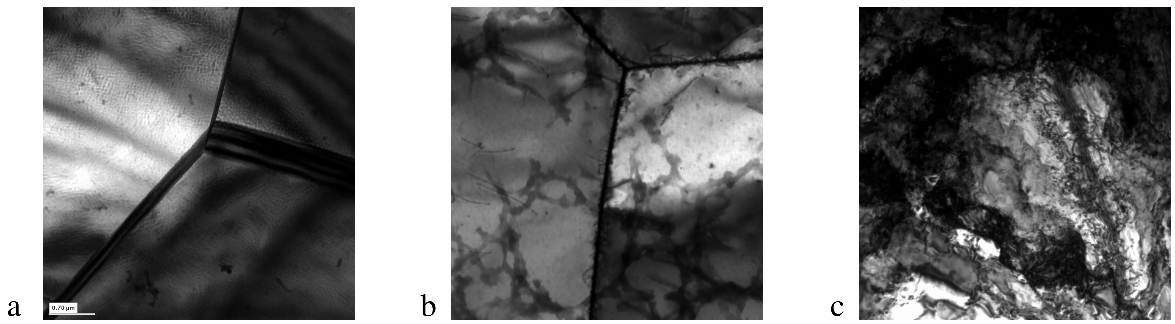

Figure 5. TEM analysis of the samples at 4.2 GPa. a) Original specimen, b) specimen loaded and unloaded with no shearing, and c) specimen loaded and sheared.

These results show that while some dislocation motion occurs near the specimen centers during loading of the specimens, the crystallites remain largely undeformed. The structure therefore must undergo primarily elastic strain during loading to high pressures. As for the sheared specimens it is evident that dislocation activity took place (figure 5c) and the mechanical properties were changed as evidenced by the increase in hardness.

CONCLUSIONS

The details of a new procedure to study the mechanical properties of materials deformed by shearing strains while maintained under high pressure have been described. Based on the results exposed here, this procedure has proven to be a good method to study the shear stress – shear strain behavior under hydrostatic pressure. It was observed that the microstructure and properties do not change significantly due solely to the effect of the pressure applied. This was corroborated via Vickers hardness characterization, with the hardness being almost equal to that of the un-deformed material. In addition, validation was done through structural analysis via TEM imaging. Neither of these techniques revealed significant deformation of the microstructure nor dislocation multiplication during the loading process.

The outcome of the tests showed a more influent role of the hydrostatic pressure on properties such as yield strength, hardening than that predicted by the Steinberg-Guinan model

[9,10]. The model predicts an increment in yield strength of close to 3% up to a pressure of 4.2 GPa. Experimentally, an increase close to 180 % was observed for this material at the same pressure. These results are in qualitative agreement with those obtained by Bridgman and by Weir [11]. A more extensive analysis will be given in a later paper.

ACKNOWLEDGMENTS

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REFERENCES

1. J.L. Lewandowski and P. Lowhaphandu, "Effect of hydrostatic pressure on mechanical behaviour and deformation processing of materials on materials", Int. *Mat. Reviews*, v **43**, n 4:145-187, 1998.
2. PW Bridgman, "Shearing phenomena at high pressures", Proc. Am. Acad. Arts Sci. **71**: 387-460, 1937.
3. Bridgman, P.W. 1935 "Effects of shearing stresses combined with high hydrostatic pressure", Phys. Rev. **48**: 825-847 (1935)
4. PW Bridgman, "Effects of hydrostatic pressure on the plastic properties of the metals", Rev. Mod. Phys **17**: 3-14, 1945.
5. PW Bridgman, "Flow phenomena in heavily stressed metals", J. Appl. Phys. **8**: 328-336, 1937.
6. Hsiung and D.H. Lassila, "Shock-induced deformation twinning and omega transformation in tantalum and tantalum-tungsten alloys," Acta Mater. **48**:4851-4865, 2000.
7. P.Söderlind and J.A. Moriarty, "First-principles theory of Ta up to 10 Mbar pressure: Structural and mechanical properties," Phys. Rev. B **57**:10340-10350, 1998.
8. Lassila, "Strength of materials under high pressure", Report LLNL.
9. DJ Steinberg, SG Cochran, MW Guinan "Constitutive model for metals applicable at high strain rate", J. Appl. Phys. **51**: 1498- 1505, 1980.
10. D. Steinberg, D Breithaupt, C Honodel "Work-hardening and effective viscosity of solid beryllium", Physica **139 & 140B**: 762-765 (1986).
11. S.R. Weir, J. Akella, C. Ruddle, T Goodwin and L. Siung. "Static strength of Ta and U under ultrahigh pressures." Phys. Rev. B **57:**11258-11265, 1998

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